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Monitoring epi-ready semiconductor wafers

D.A. Allwood, S. Cox, N.J. Mason*, R. Palmer, R. Young, P.J. Walker

Physics Department, Clarendon Laboratory, University of Oxford, Parks Road, OX1 3PU Oxford, UK

Abstract

GaAs wafers are often sold by the manufacturers as 'epi-ready', meaning the substrates can be used with no further treatment. However, there is no clear agreement what epi-ready means, although one manufacturer recommends that they be used within 3 months of purchase. However, many growers find they are unable to obtain good growth results from their processes with untreated wafers and have to resort to long-established etching procedures. With the increase in multi-wafer molecular beam epitaxy (MBE) and metal organic vapour phase epitaxy (MOVPE) reactors, this approach is no longer viable. This work investigates the ageing of GaAs wafers stored, both under a variety of conditions [e.g. temperatures of 200 °C, room temperature and –20 °C] and assessed by atomic force microscopy (AFM) after various procedures had been performed, including annealing in molecular hydrogen using a MOVPE reactor. The primary source of ageing on an epi-ready GaAs wafer has been found to be the transfer oxidation from the native As_2O_3 (present at time = 0) to Ga_2O_3 (present after 24 h, 3 months and 2 years, respectively, at the storage temperatures mentioned above). This transfer oxidation does not occur across the whole surface but in discrete areas that may be associated with the step edges on the substrate surface. This is the cause of deterioration of an epi-ready substrate, but it can be slowed by storing the GaAs wafers at –20 °C. Storage in a domestic freezer has allowed even 2-year-old GaAs wafers to be used without any further preparation. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Monitoring epi-ready; Semiconductor; Wafers

1. Introduction

Epitaxial techniques such as MBE and MOVPE rely on the perfection of the surface of the substrate to give a template that allows further growth of high quality semiconductor crystals. Semiconductor substrates are manufactured from boules of material grown from a melt by various processes [1]. The substrates are then sawn from the boule and chemo-mechanically polished. All these stages can introduce defects into the substrate: the thermal strain from growing the boule can result in dislocations in the final substrate [1], cutting can introduce 'saw-damage' into the substrate whilst polishing can leave the final surface non-stoichiometric, e.g. with an excess of one element (usually group III) over the other.

The provision of a perfect, stoichiometric surface used to be the responsibility of the grower who had to prepare the wafer before use [involving degreasing, etching (to remove saw-damage), rinsing and drying]. Now, however, there is a choice and the term 'Epi-

Ready' has been registered as a trademark of Wacker-Chemitronic GmbH [2]. This new approach was made possible by low metal contamination in the polishing and by producing a final surface whose carbon and oxide layers were removable by in situ cleaning in a MOVPE reactor. Now, many other substrate vendors are offering substrates 'epi-ready' but there is no strict definition of the term. The responsibility for the provision of a perfect surface is now a partnership, the wafer manufacturer supplying an epi-ready surface and the grower finding conditions where this can be successfully deoxidised and annealed in the equipment before growth [3,4]. With increasing wafer numbers in multi-wafer reactors, this partnership is the only realistic choice for most industrial reactors.

A study of InP substrates [5] and GaAs substrates [3] have both found an increase of the oxide thickness with time but no data was presented showing how this affected the epi-readiness. Although one manufacturer recommends that GaAs wafers are used within 3 months of purchase, there is no information in the literature about what occurs over this period of time to leave the GaAs surface unepi-ready nor under what storage conditions this degradation occurs.

*Corresponding author. Tel.: +44-1865-855167; fax: +44-1865-371090.

E-mail address: nigel.mason@kamelian.com (N.J. Mason).

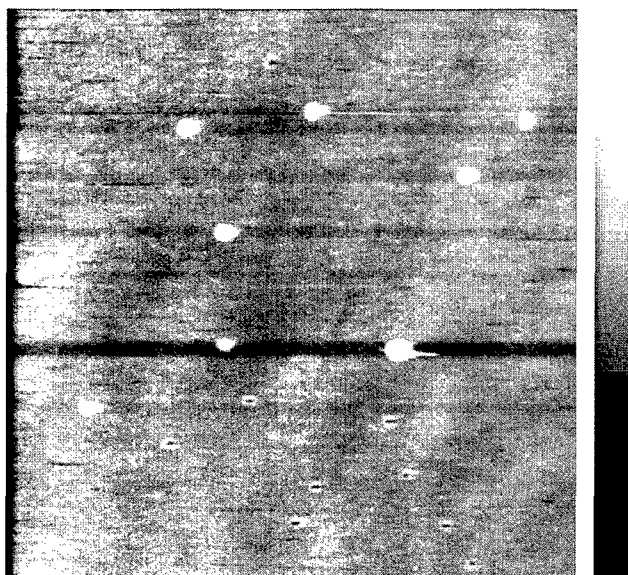


Fig. 1. Atomic force microscope image (6- μm square) of an on-axis GaAs surface after heating to 550 °C, holding and cooling. The grey scale represents 10 nm in height.

We have used a variety of techniques to study the ageing of GaAs wafers; the loss of epi-readiness and the provision of best storage conditions and the results are reported here.

2. Experimental

The wafers used in this work were obtained from AXT, Freiberger, and Wafer Technology and were on-axis. We neither observed any measurable difference between any of the manufacturers nor between doped and undoped wafers under the conditions used here. The wafers were stored in a domestic freezer (–20 °C) a domestic fridge (0 °C) normal clean room ambient (20 °C) whilst remaining in their original polyethylene packing and inert gas filled metallised-polyester bag. For elevated-temperature storage (200 °C) the wafers were removed from the packing and placed in a clean, covered Petri dish in a muffle furnace. Care was taken to ensure the door of the furnace remained open for as short a time as possible when adding and removing samples, to ensure a stable temperature regime for the storage trial.

The reactor conditions used have been outlined before [6] but to reiterate briefly a flow of 16 l/min of hydrogen at atmospheric pressure was used in these experiments. The samples were usually ramped up to 550 °C at 40 °C/min, held at temperature for 15 min, and cooled at the same rate.

Homoepitaxial growth was achieved at 660 °C using trimethylgallium (–9 °C) and tertiarybutylarsine (5 °C).

The HCl/water dip involved submerging the wafer in a 50% HCl/water solution for 30 s and then rinsing for

1 min in de-ionised water and then drying using filtered nitrogen.

The atomic force microscopes (AFM), used in this study were either a Burleigh Metris or Thermomicroscope M5 and results were similar in either case.

3. Results and discussions

Fig. 1 is an AFM image of a GaAs wafer heated up to 550 °C, held, and cooled, under molecular hydrogen. We were establishing conditions for the growth of InSb quantum dots on GaAs substrates directly (no buffer layer) and in order to avoid contaminating the InSb with arsenic, we heating up the GaAs wafers with no group V species present. Since InSb grows best at 400 °C, it seemed unlikely that the GaAs would decompose, even if heated to 550 °C before growth to de-oxidise it as we had previously established conditions for the deoxidation of GaAs using optical monitoring [6,7]. When we checked that this was correct we observed the islands in the AFM image, which are present even though nothing has been deposited. An obvious possibility was that there was some decomposition of the GaAs surface, so we undertook a series of experiments. First, we tried fresh wafers, straight from the manufacturer (Fig. 1 was obtained using a 3-month-old wafer) and no islands were observed. Secondly we treated the older wafers with an HCl dip (it is known that this dip only removes As_2O_3 and Ga_2O_3 from the wafer [8–10]) again no islands were observed. Then we varied the temperature of anneal between 450 °C and 650 °C and the features only changed in their size and number

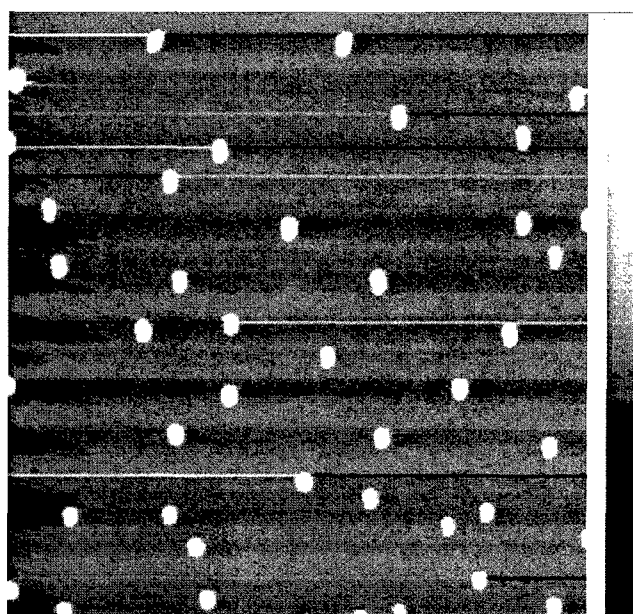


Fig. 2. Atomic force microscope image (6- μm square) of an on-axis GaAs surface after heating to 450 °C, holding and cooling. The grey scale represents 30 nm in height.

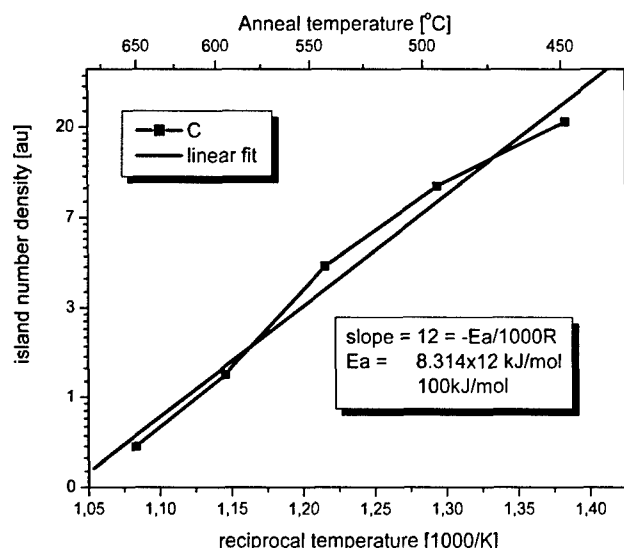


Fig. 3. Logarithmic plot of island number density vs. reciprocal absolute temperature. The grey line is a linear fit to the data.

density. As an example, Fig. 2 shows the results of heating to 450 °C and annealing.

Such a change in the number density and size of these features is indicative of a kinetically controlled surface diffusion phenomenon. In order to check this, the island area and number density are measured and calculated over multiple AFM images using analysis and counting software to give statistically sound data. A scan size of 30 μm square was initially investigated and if this proved to be free of defects and damage the area was divided into nine 10- μm squares and the number density was statistically assessed to give a mean and standard deviation, this allowed for accurate comparisons to be made between various experimental conditions and an assessment of errors and standard deviations. Fig. 3, shows the log of the number density plotted against the reciprocal absolute temperature. The linear nature of this plot means that the island growth is a kinetically controlled surface phenomenon and an activation energy of 100 kJ/mol can be calculated from the fitted data in Fig. 3. Such a value suggests that the islands grow by a surface diffusion process and we would suggest that they are islands of gallium but not due to incongruent decomposition of the GaAs, for the following five reasons:

1. The value of 100 kJ/mol (1.2 eV) is similar to that obtained (1.1 eV) from the diffusion of Ga atoms on GaAs in a MBE study [11,12].
2. An AFM study [13] involving heating GaAs wafers in a MBE chamber with no arsenic overpressure showed very similar island size and number density and was able to chemically confirm the islands as being composed only of gallium by X-ray photoelectron spectroscopy.

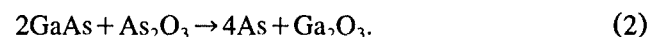
3. The temperature of the 450 °C anneal is well below that for incongruent evaporation of GaAs (650 °C) [14].
4. The islands are absent when we anneal the wafers under tertiarybutylarsine that can pyrolyse at temperatures as low as 450 °C.
5. The islands are considerably softer (when measured by lateral force microscopy, an adjunct of AFM) compared to similar sized GaSb islands.

These results were obtained, both on the traditional horizontal single wafer reactor and on our newer vertical reactor (similar to a close spaced reactor [15]) so it seems unlikely that there is some artefact from the transfer of any species from a hot susceptor upstream of the sample in the horizontal reactor.

The results suggest that age, storage, and treatment have a considerable effect on GaAs wafers when annealed under molecular hydrogen. An explanation for this lies in the nature of the native oxide present on any GaAs wafer suggested over 20 years ago [16]. Initial oxidation of a fresh GaAs wafer takes place on the arsenic sites and is indicated by the following equation:



This very rapid surface reaction quickly becomes passivated as the diffusion of oxygen through the oxide layer slows down after the growth of a few monolayers of oxide. After a few days the thickness of As_2O_3 has reached 1–2 nm (a similar thickness to that seen for oxides growing on aluminium or titanium or other self-passivating oxide systems). Subsequently, a much slower solid-state reaction takes place in between the As_2O_3 and the bulk GaAs:



This reaction occurs very slowly, as a typical bulk solid-state reaction, over a period of months. It occurs even if the wafer is stored in its original inert gas filled, metallised polyester bag, because the As_2O_3 is present on the wafer surface before the bag is sealed.

In order to examine the morphological implications of Eq. (2), a 2-year-old wafer, expected to have had a considerable amount of Ga_2O_3 formed, was examined in the AFM after an HCl/water dip. As mentioned above, the dip is expected to remove only the oxide. Fig. 4 shows a high magnification AFM of such a wafer. Significant features are observed in an otherwise flat, amorphous surface. The features are approximately 100 nm across and 4 nm deep. We suggest that these features are the result of the removal of Ga_2O_3 that had developed from Eq. (2). This idea is supported by our observation that a similar HCl/water dip on a freshly supplied substrate shows a flat amorphous surface of

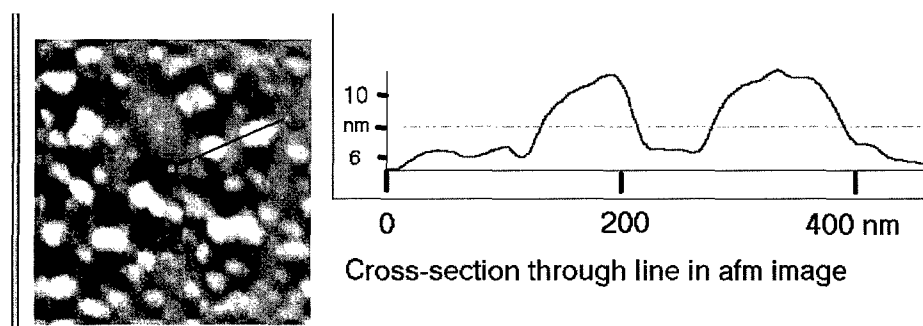


Fig. 4. Atomic force microscope image of an on-axis GaAs surface after an HCl/water dip. The image is a 1- μm square and the grey scale represents 12 nm with a line roughness of 1.9 nm.

roughness 0.5 nm. As no localised Ga_2O_3 is expected on a fresh wafer this confirms that the features in Fig. 4 are indeed from localised oxidation via Eq. (2). Fig. 5 shows a low-magnification image from the same surface as in Fig. 4. Here the pits seen in Fig. 4 are seen to be coalesced in lines running down the image. The spacing between these lines is of the order of 600 nm which is very similar to that expected for the separation of step edges on the on-axis GaAs surface used in these studies suggesting that the oxidation is somehow influenced by the step edges. Fig. 6 shows a schematic of what we propose is happening over a period of some months and after a subsequent HCl/water dip. That the features are possibly initiated at the step edges is reasonable from an energetic point-of-view as kink sites on step edges would be favourable as

places for reactions to occur. The change of the wafer surface from an atomically flat surface covered in a thin uniform layer of As_2O_3 to a much rougher surface with a thicker oxide over a period of months is in good agreement with previous X-ray diffraction and ellipsometric studies [7,17,18]. It is also likely to affect the quality of growth of any epitaxial layer. There is also the possibility that if arsine is used in the deoxidation stage before epitaxy, then the lack of pyrolysis of arsine

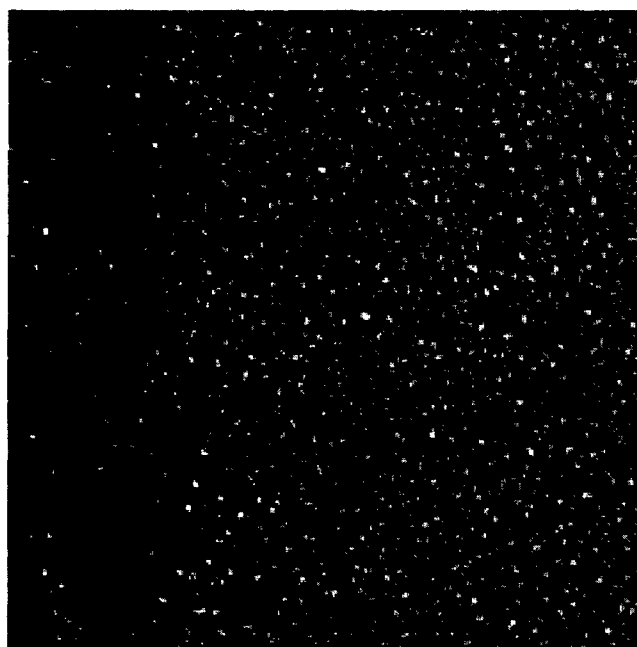


Fig. 5. Atomic force microscope image of an on-axis GaAs surface after an HCl/water dip. The image is a 10- μm square and the grey scale represents 8 nm with a line roughness of 1.2 nm.

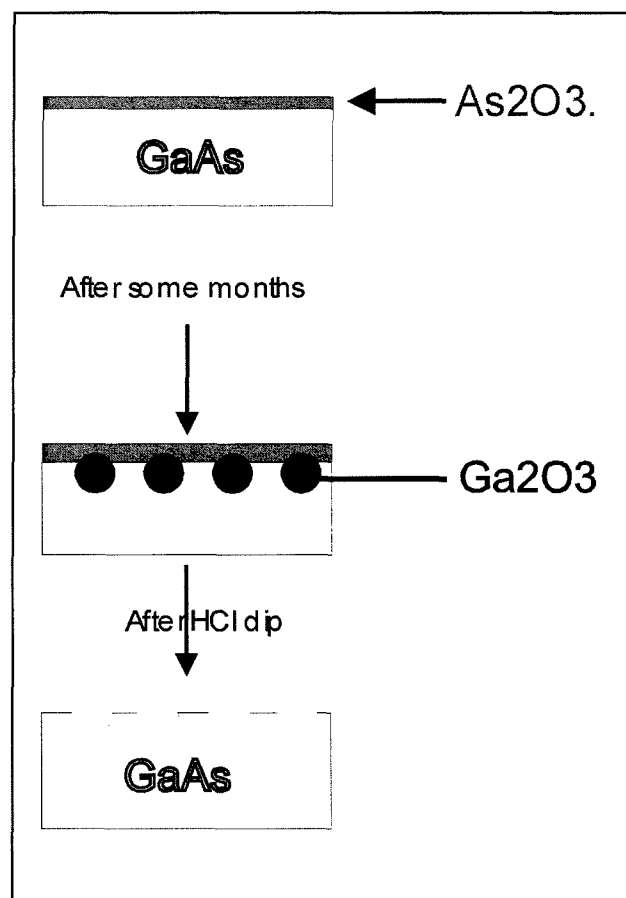


Fig. 6. Schematic of GaAs oxidation [Eqs. (1) and (2)] and subsequent surface after HCl/water dip.

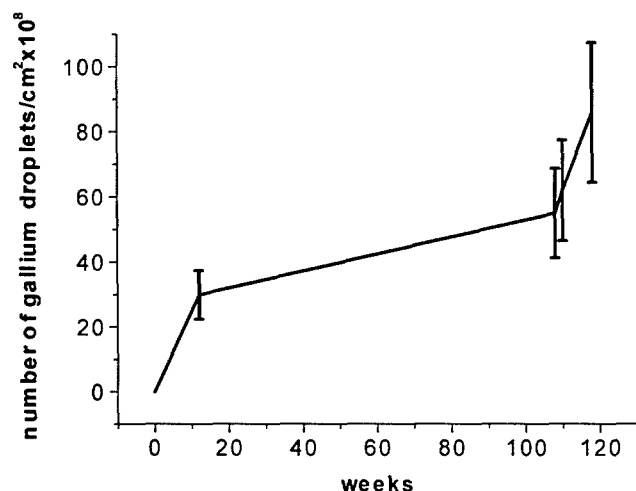


Fig. 7. Plot of number density of gallium droplets/cm² vs. time since polishing (in weeks).

at the temperatures where these islands are observed might lead to them forming before sufficient arsenic is present to mop up the gallium. This would lead to large islands of GaAs forming at the higher temperatures when the arsenic is present from pyrolysis and is absorbed into the gallium droplets. Such a surface would lead to subsequent growth having very poor morphology. Either of these morphological issues might be the reason why GaAs is only given a 3-month certificate of conformity to an epi-ready state. To study this aspect of ageing we instigated an ageing trial with wafers stored under ambient conditions and then examined them using both annealing (as above) and homoepitaxial growth.

3.1. Ageing trial

The full ageing trial is currently on going and will be reported later. Initial results confirm that the number density of the gallium droplets found under similar conditions to those used to produce Fig. 1 follow a trend that supports our above hypothesis that they are indicative of a surface that has been aged beyond 3 months. Fig. 7 shows the number density with respect to time. The error bars are quite large and we are still investigating the source of this variation but, overall, the graph agrees well with our hypothesis that even over the first month after polishing the wafer has an increasing amount of Ga₂O₃ which gradually changes the surface from epi-ready to unepi-ready, the actual time when this would manifest itself as poor growth will depend on the conditions used during the bakeout (arsine or tBAs). To show this we have used two different conditions to grow homoepitaxial GaAs as part of the ageing trial. Growth 1 is a set of typical conditions with tBAs switched into the reactor during both the heat-up and cool-down as well as during the homoepitaxy.

Growth 2 is a set of conditions that attempt to mimic arsine whilst using tBAs. To do this we have only switched the tBAs into the reactor at a temperature (500 °C) to mimic the pyrolysis of arsine [19]. When we use Growth 1 on wafers that are 0 and 4 weeks old we get atomically flat terraces (average roughness 0.1 nm) in the AFM similar to those seen Fig. 8. On a 12-week-old wafer the homoepitaxial layer no longer grows in step flow to give atomically flat terraces but in an island growth mode giving a very much rougher surface (average roughness 1.5 nm) seen in Fig. 9. This change is also seen in Growth 2 but the rougher, island growth occurs at week 4 not week 12. This would suggest that the length of time a wafer is epi-ready is determined by the precursors used and perhaps the reactor geometry and there is no specific time limit that covers all possible conditions.

Eq. (2) suggests that it might be possible to increase the rate of gallium oxidation by increasing the temperature. In order to test various post-polishing treatments we wished to develop a way of ageing GaAs over a suitable period (days) to be able to predict the results over longer periods (months). We have stored wafers at various elevated temperatures to determine if this can accelerate the ageing discussed above. We have been able to accelerate the reaction occurring in Eq. (2) by storage of the wafers at elevated temperatures for quite short periods and Table 1 shows the results. Clearly, storage at 200 °C gives similar results, in terms of gallium island density to storage at room temperature at

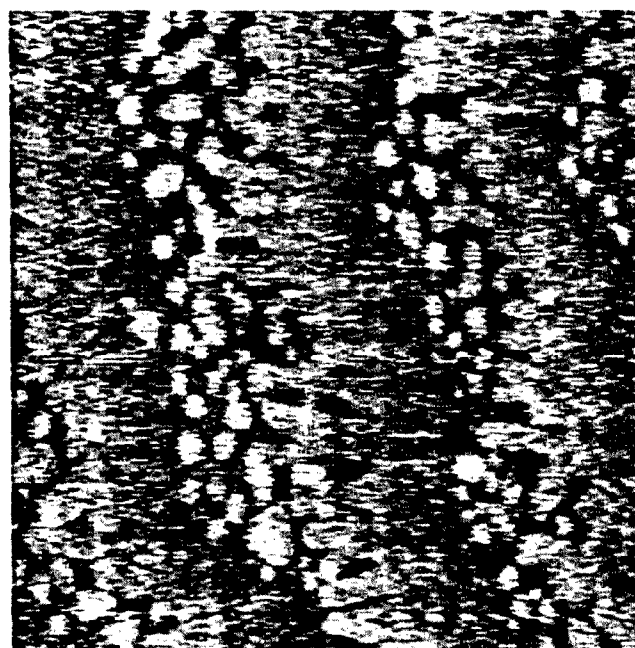


Fig. 8. Atomic force microscope image of a fresh on-axis GaAs surface after homoepitaxial growth. The image is a 3-μm square and the grey scale represents 1 nm with a line roughness of 0.1 nm.



Fig. 9. Atomic force microscope image of older on-axis GaAs surface after homoepitaxial growth. The image is a 3- μm square and the grey scale represents 10 nm with a line roughness of 1.5 nm.

20 °C for over 2 years. This facility will enable us to test rapidly any surface treatments that might be expected to improve the ageing properties of a GaAs wafer (e.g. by modifying the oxide). The results presented here are also supported by XPS measurements made at 200 °C where the reaction in Eq. (2) is observed directly [20] as a change in the peak intensities of the two oxides, the arsenic oxide diminishing and the gallium oxide increasing over a period of a few hours. This suggests quantitatively at least that temperature will play an important role in wafer storage.

An alternative, quantitative, technique for studying the progress of Eq. (2) is to use Raman scattering to monitor the signal obtained from elemental arsenic precipitated from the GaAs when it is oxidised by the As_2O_3 . The arsenic peak is easily identifiable in Raman spectra of GaAs at 260 cm^{-1} [21]. A typical GaAs Raman spectra LO phonon at 295 cm^{-1} is shown in

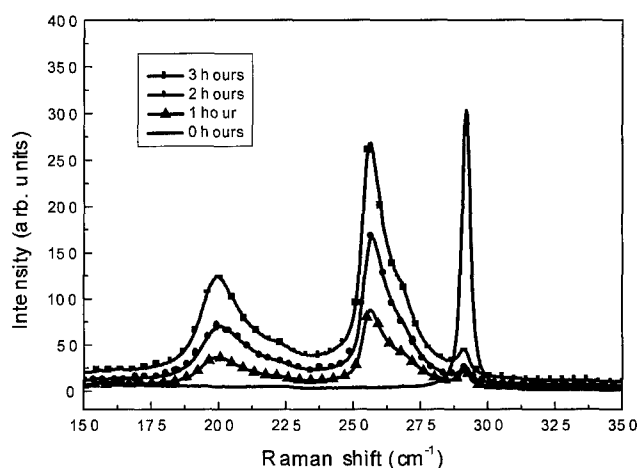


Fig. 10. Raman spectra of GaAs, fresh and after several hours at 600 °C in oxygen ambient.

Fig. 10, together with the spectra taken after high-temperature annealing in air for various times. As expected, the arsenic content of oxide layers increased with anneal time. However, surprisingly, when the ambient of the furnace was changed from oxygen to dry nitrogen, elemental arsenic content initially increased to that seen after 1 h in Fig. 10, and stopped increasing. It seems that the As_2O_3 present on the surface is enough to allow two to take place for perhaps 30 min but once the As_2O_3 is depleted further arsenic cannot be precipitated, because Eq. (1) is inhibited in the dry nitrogen ambient and hence the Ga_2O_3 ceases to form.

We also examined this elemental arsenic peak in a sample held at various temperatures between 300 and 750 °C for 1 h. The amount of elemental arsenic present increased with temperature. Fig. 11 shows an Arrhenius plot of the integrated arsenic Raman peak intensity as a function of temperature. Calculations from curve fitting constants provide an activation energy, for the formation of elemental arsenic of 0.46 eV. It should be stressed

Table 1
Number density of gallium droplets for various storage times/temperatures

Sample	Temperature of storage (°C)	Time of storage	Number of Ga droplets ($\times 10^8 \text{ cm}^{-2}$)	S.D. ($\times 10^8 \text{ cm}^{-2}$)
Stored	20	24 months	1.8	0.1
Aged	100	3 weeks	0.6	0.1
Aged	200	20 h	1.6	0.1
Aged	200	12 h	0.5	0.05
Stored	−20	24 months	0.0	

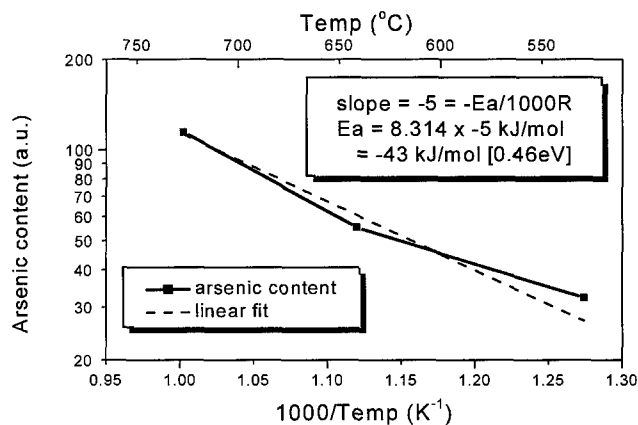


Fig. 11. Logarithmic plot of arsenic content vs. reciprocal absolute temperature. The grey line is a linear fit to the data.

Table 2

Estimated relative ageing rate of GaAs surface oxides for different storage temperatures using data from Fig. 11

Storage temperature (°C)	Relative rate of ageing	Age at loss of epi-readiness (months)
20	1.00	3
0	0.26	12
–10	0.13	24
–20	0.06	48

that this is an approximate value since there are only three data points present and the values of each of these are subject to considerable uncertainty. However, it is in good agreement with values in the literature [21].

These two results from the Raman measurements have two important implications for the storage of GaAs wafers. Firstly, the activation energy suggests that storage at reduced temperatures might lead to the slowing of the reaction in Eq. (2) and thus a slowing in the degradation of the GaAs wafer. Secondly, storage in an inert gas filled bag will not, of its self stop Eq. (2) taking place and the wafer will degrade until all the As_2O_3 has been consumed, it will then stop. Table 2 shows approximate values for the decrease in rate of ageing by storage at various temperatures. These data suggested to us that storage in a domestic freezer was worth investigating as a means of enhancing the life expectancy of an epi-ready wafer. We stored two wafers under ambient conditions, two at 0 °C in a domestic fridge and two at –20 °C in a domestic freezer. Two years later, the wafers were removed from the freezer and assessed by annealing as above. No evidence of gallium droplets was observed for those wafers stored at –20 °C suggesting that the above hypothesis was sound. These wafers are being assessed by other techniques such as growth and the results, which look very encouraging, will be reported with the final results of the ageing trial.

4. Discussion

One of the difficulties in assessing the literature on investigations into the thickness and nature of the oxides found on GaAs is that often there is no information as to the conditions under which the wafers have been stored or prepared. Discrepancies between various authors concerning both the thickness and the type of oxide present could be accounted for by the age of the sample or by the way it was prepared. An oxidative etch such as sulfuric acid/hydrogen peroxide [22] leaves the surface with Ga_2O_3 present and will affect any surface studies compared to an epi-ready surface [22] which will initially have little Ga_2O_3 if prepared under an alkali environment [7,18].

Substrates that have been stored for longer than 3 months can be re-generated by dipping in HCl/water but this is nearly as time consuming as etching in sulfuric acid/hydrogen peroxide and so cannot be recommended as a way of avoiding the problems of a unepi-ready wafer. Instead, storage in a domestic freezer has been found to be highly successful in preventing the degradation associated with Eq. (2). This is of particular use where substrates have to be bought in large quantities to save money but then would otherwise languish on a shelf for several months and deteriorate and prove a false economy. Provided the wafer is removed from the freezer 1 h before it is needed, to allow the temperature inside the polyethylene packaging to equilibrate with the ambient (thus preventing any condensation) we have seen no deleterious effects by storage under these conditions.

We have not seen any difference between doped and undoped GaAs in the measurements made here, despite the fact that subtle differences have been reported [23]. We have found some differences when looking at vicinal substrates (specifically 2° off towards [111]A) compared to the on-axis substrates studied here but these will form part of our next report on the full ageing trial.

5. Conclusions

Epi-ready GaAs wafers have been stored under a variety of conditions with temperatures between 200 and –20 °C and assessed by measuring the surface in an AFM after various tests including annealing in molecular hydrogen using a MOVPE reactor.

The primary source of ageing on an epi-ready GaAs wafer has been found to be the transfer oxidation from the native As_2O_3 (present at time=0) to Ga_2O_3 . The kinetics of this equation has allowed us both to enhance it as a means of accelerated ageing, and attenuate it as a means of storage over a period of 2 years (at –20 °C) that allows the wafer to be used as if it were freshly polished.

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